I. A NEW TECHNIQUE OF X-RAY MICRORADIOGRAPHY

II. QUANTITATIVE EVALUATION OF CRYSTALLINE DISTORTION AND FATIGUE BY X-RAY DIFFRACTION

BY

WILLIAM McKINLEY SHAFER

B.S., University of Illinois, 1934

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AN ABSTRACT OF A THESIS

SUBMITTED IN PARTIAL FULFILLMENT OF THE REQUIREMENTS FOR THE DEGREE OF DOCTOR OF PHILOSOPHY IN CHEMISTRY IN THE GRADUATE SCHOOL OF THE UNIVERSITY OF ILLINOIS, 1940

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I

A NEW TECHNIQUE OF X-RAY MICRORADIOGRAPHY

Radiography is the oldest branch of X-ray science. Hitherto, it has been limited to the study of objects large enough to give radiographic images of sufficient size to be viewed by the naked eye. Obviously, it would be advantageous to obtain and view radiographic images of heterogeneous objects of very small size.

In this investigation a technique of X-ray microradiography has been developed for the study of small inhomogeneities such as cracks, inclusions, alloy constituents, and voids in metals. The technique has been applied to the study of copper-aluminum alloys, brass, type metal, graphitic steel and small welds.

Some of the advantages found for the technique over microscopical methods are:

1. A three-dimensional view is obtained.
2. Internal cracks and other voids are readily detected.
3. The components may be differentiated by their atomic numbers.
4. Fine polishing is not required.

II

QUANTITATIVE EVALUATION OF CRYSTALLINE DISTORTION BY X-RAY DIFFRACTION

Metallurgists and engineers have frequently criticized X-ray investigators of crystalline distortion for their lack of quantitativeness. The purpose of this work was to carry on further investigation of distortion and to state the results in quantitative terms where possible.

A. X-RAY STUDY OF ELECTRODEPOSITED NICKEL FILMS

The increasing interest in metal deposits, especially bright deposits and codeposited allows combined with the empirical state of the science of electroplating and the feeling that the properties of an electro-

deposited metal depend upon its structure, caused a part of this investigation to be devoted to an X-ray diffraction study of the structure of nickel deposits deposited under various conditions. Some of the X-ray diffraction investigations in the field of electrodeposition are those of Glocker and Kaupp[1], Wood[2,3], Palatnik[4] and Bozorth[5].

Synthetic fiber patterns were constructed for the (200) and (111) planes and for [100], [110], [111], [210] and [211] fiber axes. The type of fibering present in the deposits was then identified by comparing the diffraction patterns registered at a grazing angle of 18 degrees and at a distance of 1.5 centimeters with the synthetic patterns. The degree of fibering was measured by taking densitometer readings of maxima and minima of the 200 and 111 lines on the reflection patterns. The ratio of these readings was taken as a measure of the degree of fibering.

Specimens were rated as to particle size by a visual inspection of the patterns. The brightness of each deposit was measured by the use of a Keuffel and Esser color analyzer.

A definite correlation between brightness, on the one hand, and grain size and orientation on the other was found. The brightest deposit was the one possessing a combination of small grain size and orientation to the highest degree. The dullest deposits were those possessing a rather large grain size and very little or no orientation.

B. QUANTITATIVE EVALUATION OF DISTORTION IN SILICON STEEL AND IN ALUMINUM

Distortion or strain in crystals is manifested in the X-ray diffraction pattern by radial streaks or asterism; by radial widening of lines; by shifting of lines and spots; by peripheral widening of spots; and by changes in intensities of reflections. Any quantitative work must correlate one or more of these pattern manifestations with stress or strain.

Leonhardt[6] calculated the curves along which the Lane spots should elongate for a given axis of bending and crystal orientation. Regler[7] measured the peripheral widening of spots as a function of the amount of deformation and claimed great accuracy for this method. Clark and Beckwith[8] devised a modulus based upon the ratio of the longest dimension of a Laue spot divided by its shortest dimension which served as a measure of slight residual distortion in silicon steel,

In this investigation single crystals of silicon steel were bent

measured amounts by known stresses. The bending of the crystal was correlated with the elongation of the Laue spots measured in degrees. Elastic elongation of the Laue spots was found to occur within the elastic limit of the specimen. Whether the resulting "radial asterism" was due to elastic bending of planes or rotation of "blocks" was not decided.

Nadai's[9] octahedral shear equations were used for expressing the cold work produced by rolling and elongation of both polycrystalline and single crystalline aluminum. For equivalent amounts of cold working, as expressed by Nadai's octahedral shear, the effects of cold rolling and elongation, within the limits of deformation studied, upon the X-ray pattern were found to be nearly the same. The patterns showed marked resemblance when examined visually and when quantitative measurements of the elongation of the Lane spots in degrees were made.

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